

# Selective froth flotation concentration of antimonite ore from a mixture of antimony, lead and arsenic ores

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**Abstract**— Mineral ore beneficiation and value addition has become a key factor for economic growth in Africa. Froth flotation process is used to concentrate relatively low grade ores. In Zimbabwe antimonite is mined together with lead and arsenic which are regarded as contaminants and hence the need to selectively remove them. We report on the selective concentration of antimonite at the expense of lead and arsenic by use of a depressant. Starting with an containing antimony (5.89%), lead (0.34%) and arsenic (0.25%) a concentrate containing antimony 30.68%, lead 1.52% and arsenic 0.85% was obtained without an depressant in a Devnver flotation cell. The addition of a depressant resulted in a concentrate with antimony 24.89%, lead 0.90% and arsenic 5.89%. The Flame Atomic Absorption Spectroscopy (FAAS) and X-Ray Florescence (XRF) analytical techniques were used to measure the changes in concentrations of antimony, lead and arsenic during this research.

**Index Terms**—antimonite, arsenic, froth flotation, lead.

## I. INTRODUCTION

The froth flotation process is used for ore concentration by utilizing the physio-chemical differences among mineral particles. The process comprises of chemical treatment on the surface of the mineral ore in slurry to produce favorable conditions on the surface for attachment of mineral particles to air bubbles [2]. The mineral composition of the ore sample from Gothic mine Zimbabwe is an antimonite ( $\text{Sb}_2\text{S}_3$ ). The antimony is found in many forms, mainly antimonite, much subordinated in lead-antimony sulphosalts, in compounds with gold (auroantimonite) and in oxides (kermesite, valentinite). The lead is concentrated as monosulphide (galenite) and subordinated in lead-antimony sulphosalts. Arsenic is bounded mainly in arsenopyrite and absolutely subordinatedly in secondary carbonate.

Froth flotation relies on the surface of the valuable mineral being hydrophobic while the surface of the gangue minerals is hydrophilic. Equilibrium is achieved between the solid-air, liquid-air and solid-liquid interfacial tensions are in contact with solid, liquid and gas phases [3]. A hydrophobic surface is easy to float since it favour contact with air over water due to a lower free energy and hence will readily stick to an air interface if one is available. Adsorption plays an important role in the relationship between the chemical reagents in flocculation and frothers and collectors in flotation. The flotation process requires the adsorption of chemicals onto the

mineral surface to be performed selectively so that only the valuable mineral surface becomes hydrophobic while the gangue mineral surfaces become or remain hydrophilic [4]. Most minerals are not water-repellent in their natural state and flotation reagents must be added to the pulp. Generally the most essential reagents in flotation process are the collectors, which are used to adsorb on mineral surfaces, rendering them hydrophobic and facilitating bubble attachment [5]. The frothers are used to maintain a reasonable stable froth during froth flotation. Regulators helps to control the flotation process, which are either activate or depress mineral attachment to air bubbles and are also used to control the pH of the system[2].

The knowledge obtained from this research will be a step forward for Gothic mine in fulfilling the laws and regulation of Zimbabwe by beneficiation and value addition of antimony ores before export [1]. The research will bring enlightenment on the extent to which antimony ore can be concentrated while suppressing lead and arsenic by employing a flotation process.

## II. MATERIALS AND METHODS

Potassium hydroxide (Cosmos), Lead nitrate (Associated Chemical), copper sulphate (Sky labs), Hydrochloric acid (Sky labs), tartaric acid (Associated Chemical), Nitric acid (Sky labs), perchloric acid (Sky labs), potassium amyl xanthate (Semmin), Sodium silicate ( Sky labs), Sodium Cyanide ( Sky labs), Potassium dichromate ( Sky labs), Sodium isobutyl xanthate (Senmin), Methyl isobutyl carbinol (Senmin), Polypropylene glycol ethers (Senmin), Flame Atomic Absorption Spectroscopy (55B AAS, Varian), X ray florescence (Niton XL3 Thermoscientific), Denver machine (D12, Siemens), Vibratory Sieve Shaker (AS 450 Retsch), Pulveriser (GM/F2000-B, Shibang mechner)

## III. PROCEDURES

### Sample collection

Antimonite ore was collected at from Gothic Mine in Siloba district, Lower Gweru, Midlands Province, Zimbabwe. The sample technique used to collect the ores at each stock pile was measuring two meter apart and 30cm deep. A total of ten samples were collected and labelled to represent the ten stock piles at the Gothic Mine.

### Ore liberation and comminution

The mineral ore was crushed using jaw crushers and then grinded by grinders and milled using ball mills to sizes of 70% passing 75 microns. For each sample, coning and quartering method was used to homogenous and reduce the sample size respectively.

### Effect of particle size

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To determine the optimal size for froth flotation of the antimonite, three samples weighing 1kg each with particle size ranging from 80% passing 75µm, 80% passing 150 µm and 80% passing 220 µm respectively were transferred into a Denver cell. Water was then added and the mixture agitated for 3 minutes by stirring before 1g of  $\text{Pb}(\text{NO}_3)_2$ , 1g potassium amyl xanthate (PAX) collector and 4drops of methyl isobutyl carbinol (MIBC) frother were added were in that order. The run was repeated 3 times.

### Effect of pH

The effect of pH was determined by varying from 4 to 11 using either Lime( $\text{CaO}$ ) or 0.1 M  $\text{HCl}$ . After agitating for 3 minutes as described above; 1.0g  $\text{Pb}(\text{NO}_3)_2$ , 0.5g PAX collector and 4drops of (MIBC) frother were added.

### Effect of frothers

To determine the effect of frothers two types of frothers were tested, that is, methyl isobutyl carbinol (4drops) and polypropylene glycol ethers (4 drops). Other conditions were kept constant as follows; 1.0g  $\text{Pb}(\text{NO}_3)_2$  and 0.5g (PAX) collector were added.

### Effect of activators

To determine the effect of activators 1.0g of  $\text{CuSO}_4$ , 1.0g  $\text{Pb}(\text{NO}_3)_2$  and 1.0g  $\text{BaCl}_2$  were tested individually with. 0.5g potassium amyl xanthate (PAX) as collector and 4drops polypropylene glycol ethers (senfroth) as frother were added to a 1 kg of sample as described above.

### Effect of promoter

0.5g PAX collector, 1.0g  $\text{Pb}(\text{NO}_3)_2$  activator, 0.5g of Sodium isobutyl xanthate (SIBX) promoter and 4drops polypropylene glycol ethers (senfroth) frother were used.

### Effect of varying collector and activator simultaneously

$\text{Pb}(\text{NO}_3)_2$  activator dosage was increased from 1.0 to 2.0g. PAX collector dosage increased from 0.5g to 3.0 g and SIBX collector is increased from 0.5g to 2.0g. 4 drops of senfroth are used for each test work.

### Effect of using Potassium dichromate and sodium cyanide as depressants

Activators, collectors, promoters and frother dosage were maintained from the optimum conditions. Depressants  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{Na}_2\text{SiO}_3$  and  $\text{NaCN}$  were used and their dosages increased from 1.0g to 2.0g respectively.

### Effect of depressants

Potassium dichromate 0.5g, sodium silicate 0.5g and sodium cyanide 0.5g depressants were used. 0.5 PAX collector, 1.0g  $\text{Pb}(\text{NO}_3)_2$  activator, 0.5g of Sodium isobutyl xanthate (SIBX) promoter and 4 drops polypropylene glycol ethers (senfroth) frother were added.

### Determination of concentration of antimony, lead and arsenic

Change in concentration of antimony, lead and arsenic was analysed by using a portable XRF( ) or FAAS ( ). For XRF analysis, the samples were pulverised for 60 seconds ( size ) to increase sensitivity during analysis. FAAS involves wet methods hence the samples were first digested before analysis.

### Digestion of antimony and lead for FAAS analysis

Antimony and lead are prepared in the same matrix for AAS analysis. 0.5g sample was fused with  $\text{KOH}$  in a zirconium crucible to cherry red melt. After cooling it was leached into a 400ml beaker with 100ml distilled water and boiled to dissolve the melt. The crucible was washed, added 30ml  $\text{HNO}_3$  and 10ml of 50% tartaric acid. If there is an undissolved sulphide the solution is boiled until they dissolve.

### Digestion of arsenic for FAAS analysis

1g sample is weighed into a 250ml Phillips beaker and a mixture of 20ml 1:1  $\text{HNO}_3$  and  $\text{HClO}_4$  added. The solution is fumed to dryness. 10ml  $\text{HClO}_4$  and 30 ml water are added. The mixture is digested on low heat for 20minutes on a hot plate. The solution is cooled and transferred to a 100ml volumetric flask for AAS reading.

## IV. RESULTS AND DISCUSSION

### Effect of particle size

Particle size considerations is an important parameter during flotation process. It determines the degree of liberation of mineral from gangue and floatability of the mineral in having to overcome the gravitational force as it ascend to the surface. Each mineral will respond differently to size reduction and hence the need to determine the best particle size for each ore.

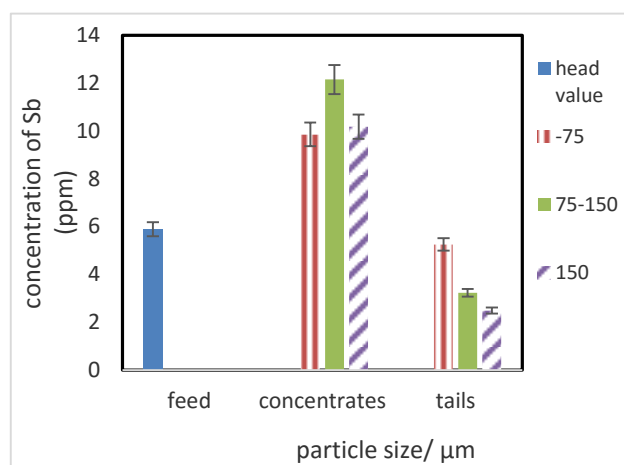
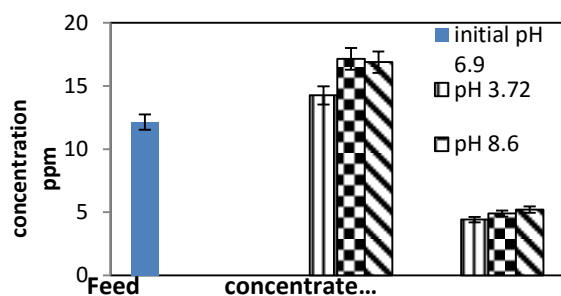


Figure 1.0 Effects of particle size on the recovery of antimony from ore.

There was a marked increase in recovery of Sb from the ore which had an initial concentration of 5.89% Sb and was beneficiated to 12.5% on the 80% passing 75-150 µm size range compared to the control which was not screened.

### Effect of pH

Pulp pH plays a very vital role in froth flotation, because the activities of minerals and reagents used are pH dependent. Reagents concentration and pH involves a very complex relationship [1]. The most ideal flotation conditions are in alkaline medium which minimizes the acidic corrosion associated with low pH.



**Figure 2.0** Effects of pH on the recovery of antimony from an ore. The solid bar shows recovery at neutral pH of 6.9. The concentrate samples showed an enrichment of Sb with the highest at pH 8.6.

The optimum recovery was recorded at pH 8.6 and it appears like there is no marked increase of Sb recovered at higher pH values as shown in figure 2.0. As expected acidic conditions are less favourable for antimony concentration because of the instability of xanthates ( $pK_a$ ) collectors at low pH values.

### Combined effects of collectors, activators, frothers, promoters and depressants

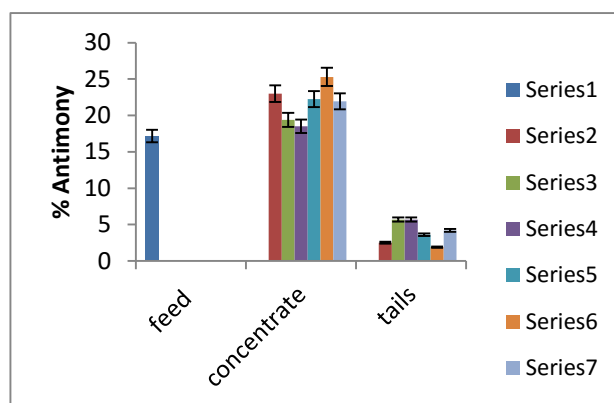
Variations of reagents; collectors, activator, frothers, promoters and depressants play a big role in selective and absorption during flotation. In order to determine the optimum combination of collectors, activators, frothers, promoters or depressants various reagents were utilized as shown in table 1. The performance of each combination was evaluated by the beneficiation of the antimony in the concentrate compared to the tailings.

**Table 1:** A combination of reagents used to determine the optimum conditions for the concentration of antimony at the expense of lead and arsenic.

Test #	Collector	Activator	Frother	pH regulator	Promoter	Depressant
1	Potassium Amyl Xanthate 0.5g	Lead Nitrate 1.0g	Methyl isobutyl carbinol (MIBC) 4drops	Lime 2.0g		
2	Potassium Amyl Xanthate 0.5g	Lead Nitrate 1.0g	Senfrother 4drops	Lime 2.0g		
3	Potassium Amyl Xanthate 1.0g	Copper (II) Sulphate 1.0g	Senfrother 4drops	Lime 2.0g	Sodium Isobutylxanthate (SIBX) 0.5g	
4	Potassium Amyl Xanthate 1.0g	Lead Nitrate 1.0g	Senfrother 4drops	Lime 6.0g		Sodium Silicate 0.5g, Sodium Cyanide 0.5g
5	Potassium Amyl Xanthate 1.0g	Barium chloride 1g	Senfrother 4drops	Lime 2.0g	Sodium IsobutylXanthate (SIBX) 0.5g	Potassium dichromate 0.5 g
6	Potassium Amyl Xanthate 1.0g	Lead Nitrate 1.0g	Senfrother 4drops	Lime 2.0g	Sodium IsobutylXanthate (SIBX) 0.5g	
7	Potassium Amyl Xanthate 1.0g	Lead Nitrate 1.0g	Senfrother 4drops	Lime 2.0g		Potassium dichromate 0.5 g

The effects of various reagents on antimony concentration during flotation tests are shown in Figure 3.0. Tests 1 and 2 compares the use of methyl isobutyl carbinol (MIBC) and

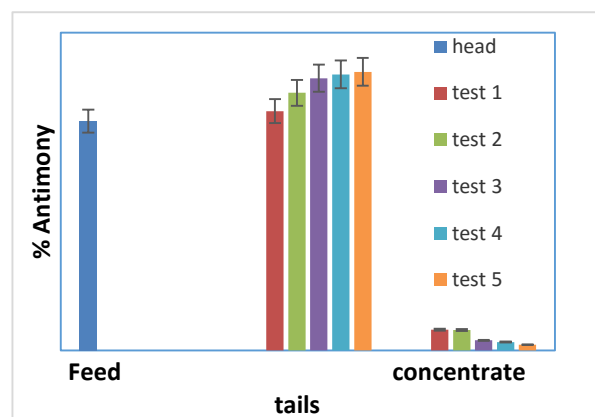
Senfrother as frothers and results indicate that Senfrother performs much better. Senfrother was used for the rest of the tests. Comparison of tests 3 and 6 show that lead nitrate is preferred activator than copper (II) sulphate in the presence of sodium isobutylxanthate. The addition of potassium dichromate as a depressant did not have benefits to the recovery of antimony as shown in tests 2 and 7. The addition of sodium silicate and sodium cyanide (test 4) as lead and arsenic potential depressants did not improve the recovery of lead. The optimum conditions were obtained when potassium amyl xanthate, lead nitrate, Senfrother, lime and sodium isobutyl xanthate were used which gave a recovery of 25.29%.



**Figure 3.0** Effects of a combination of reagents on the recovery of antimony.

### Effect of concentration of multiple reagents on $Sb_2S_3$ recovery

Optimisation tests were carried out to measure how much of antimony concentrate can be recovered during flotation. It can be deduced from the optimisation tests that increase in dosage of reagents such as collector, activator, frother and promoter results in increase of recovery of antimony. The results are summarised in figure 4.0 below:



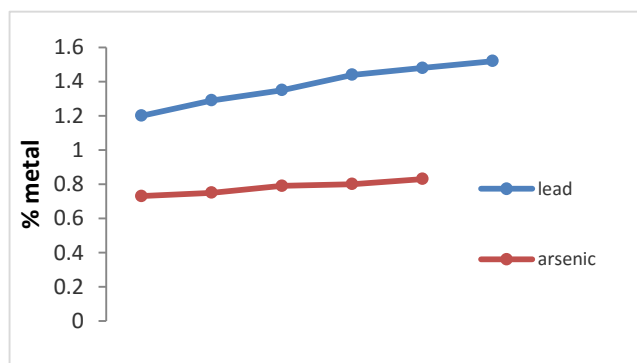
**Figure 4.0:** Optimization of antimony recovery by adjusting concentration of reagents.

From figure 4.0 recovery of antimony increased from 25.25% to 30.68%. However the change in the recovery of antimony is small from test 4 to test 5. The change in concentration is only 0.28%.

### Monitoring of lead and arsenic in concentrate

Since antimony was being selectively targeted for enrichment during flotation, lead and arsenic are expected to decrease in

concentration. However from the results obtained the increase in antimony also results in the increase of lead and arsenic as shown in figure 5.0.

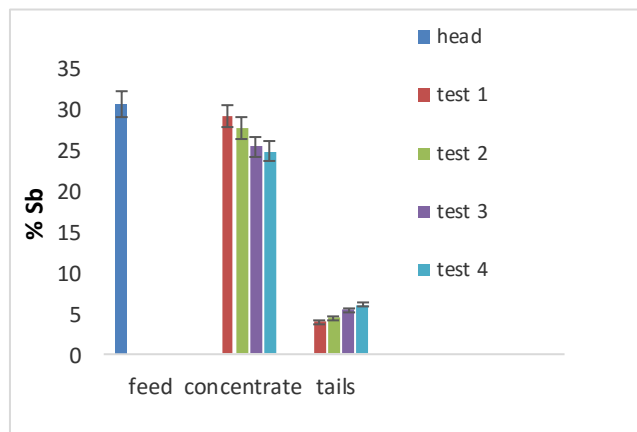


**Figure 5.0.** Change in lead and arsenic concentration with the increase in reagent concentration (c.f figure 4.0).

The lead concentration increased from 1.20% to 1.52% and arsenic increased from 0.73% to 0.83% during optimisation tests. The increase in concentration of the two metals is undesirable because of the penalty that is charged to the suppliers of the combined concentrations exceed 2%.

#### Selective depression of As and Pb

Tests were carried to determine conditions that will result in the selective depression of lead and arsenic without compromising on the recovery of antimony. The results in figure 6.0 show a noticeable decrease in the targeted antimony when depressants were introduced from a value of 30.68% to 24.89% with a corresponding increase in the tails from 3.85% to 6.01%.



**Figure 6.0.** Change in antimony concentration with increase in dosage of depressants

The addition of depressants resulted in decrease in lead and arsenic concentration values as shown in. Figure 7.0, although the decrease of the two metals are much lower than for antimony indicating some degree of selectivity between the three metals.



**Figure 7.0** lead and arsenic concentrations after Lead decreased from 1.52% ( ) to 0.90% while arsenic decreased from 0.85% (concentrate without depressant) to 0.65%.

However compared to the ore there is a less than threefold increase in lead and arsenic. The results showed a positive effect on the beneficiation of antimony at the expense of lead and arsenic.

#### IV. CONCLUSIONS AND RECOMMENDATION

The research showed that froth flotation is an effective method of selectively increasing antimony concentration while suppressing lead and arsenic. Low grade antimony ores containing concentration value of 5.89% increased to 24.89% after flotation tests. Lead concentration increased during flotation from 0.34% to 1.5% during optimization of antimony, and then lowered to 0.90% after depressant tests. For arsenic, the concentration increased from 0.25% to 0.85% during antimony optimization tests then decreased to 0.65% after depressant tests. This trend was expected from literature since optimization tests had no depressants added therefore no selective during flotation hence increased in both antimony, lead and arsenic values. Introduction of depressants in the flotation process resulted in decrease in lead, arsenic and antimony concentrations. Depressants were expected to only decrease concentrations of lead and arsenic but they also affected the recovery of antimony. However the increase in concentration of antimony was significant during the flotation process while lead and arsenic concentrations were depressed to below 2.00% lead and arsenic concentrations combined. This shows that the flotation process is very useful in solving the challenges being faced at Gothic mine of low grade antimony values with high lead and arsenic values.

Further research on optimisation tests is required by investigating other depressants. This research was carried out using a batch process but it would be important to further investigate the flotation process of a continuous process.

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